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## ACHIEVEMENT OF NODULES IN DUCTILE IRON HAVING SULPHUR CONTENT NOT LESS THAN 0.07% WEIGHT

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**Abstract:** Scraps melted were analyzed with optical light emission spectrometer for chemical composition. The charge was prepared theoretically with carbon percent of 3.9 and silicon of between 2.1 and 2.8 percent after inoculation. 2 kg of CaCO<sub>3</sub> (flux) was charged to aid removal of slag. The charges were heated to a tapping temperature of 1430° C using inserted thermocouple to monitor the temperature. The melt was desulphurised with 0.30 kg calcium carbide (CaC<sub>2</sub>) after the attainment of 1430°C. Three tappings were made tagged A, B and C into the prepared ladle via sandwich process. 3.5 %, 3.0 % and 2.0 % of ladle capacity (15 Kg) was the quantity of Mg used to treat melts A, B and C respectively. The treated melts were poured into the prepared sand moulds forming desired casting. The as-cast were analyzed again to determine its chemical compositions and found that carbon and silicon were not less than 2.0 and 1.0 % respectively. The casting was tested for hardness, tensile and micro structural evaluation. The microstructures revealed that as-cast tagged A and B formed nodular iron when etched with 2 % nital even with residual sulphur content of 0.075 % and 0.070% for A and B respectively. Cast produced from melt C revealed not a clear nodular cast iron. Emphasis is on casting A and B as we obtained nodules in A and B.

**Keywords:** Nodules, Sandwich, Sulphur Content, Metallographic Examination, Ductile iron, Rotary furnace

### 1. INTRODUCTION

Ductile cast iron is an important construction material within the cast iron family (Rikard kallbom et al, 2005). Ductile cast iron provides a wide spectrum of mechanical properties that can be obtained either by altering certain processing variables, or through various heat treatments which present different and better combination of properties for application with special requirements (Kenawy et al, 2001).

Some of the advantages offered by ductile cast iron are; good castability, low manufacturing and material cost as well as their good machinability. These advantages have led to expansions of its applications throughout the manufacturing industry (Oyetunji and Omole, 2011). Ductile iron has significantly good combination of tensile strength, ductility and toughness, along with good wear resistance and hardenability. Because of even distribution of graphite nodules and the variations in the matrix structure, the mechanical properties of ductile cast irons vary in a wide range of values (Oyetunji and Omole, 2011).

The family of ductile cast iron covers a wide range of mechanical properties, replacing successfully both cast and forged steel and malleable cast irons in many applications (such as wheels, gears, crankshaft in cars and trucks (Dicocco et al, 2010).

Ductile iron is made by treating liquid iron of suitable composition with magnesium before casting (Brown, 1994). The magnesium promotes the precipitation of graphite in the form of discrete nodules instead of interconnected flakes. As a result of the nodules formed, the material has high ductility, thereby allowing casting to be used in critical applications such as automotive transmission components e.g. sprocket, crankshaft and connecting rod (Brown, 1994).

The aim of this work is to produce ductile iron with rotary furnace that the mode of firing encourages the pickup of sulphur by the metal, and in which it is only low purity grey cast iron that can be utilized as scrap.

In developing nations having power challenges, acquisition of induction furnaces noted with high degree of melt cleanliness is not easily attainable. However, rotary furnace used for this research work is fabricated for the purpose of melting iron but can only utilize diesel fuel. Also composition controlled pig iron which can be melted as base metal for ductile iron production is not available but grey cast iron scrap. It therefore became a problem for iron 'melter' to obtain nodules in the ductile iron needed by them. This is because high sulphur (up to about 0.13 %) is present in the scrap and pick up of sulphur is imminent in the course of using diesel fuel for melting, so the 'nodulariser' would have undergone desulphurization before nodularization effect starts to take place.

Significant of the study hinges on the fact that ductile iron has become a necessity in the industrialized nations due to its numerous properties, among which are: ductility, good castability, low cost of production and possibility of structural manipulations. Achievement of nodules from low purity scraps and using diesel fired rotary furnace to produce ductile iron will help small and medium scale enterprises to achieve and sustain industrial growth especially in the developing nations.

The limitations of the study and the results obtained are in: the area of elemental loss in the furnace- in the course of the research work carbon oxidation was projected at 10% loss but was discovered that it went beyond 30 % loss depending on the time spent to hold the melt in the furnace after it has been superheated. With this, composition control may be difficult. Also as a result of utilization of diesel fuel by the furnace used, increase in sulphur pick up in the melt is a sure phenomenon; the charges, fuel and product of combustion are all in contact with each other in this system.

## 2. MATERIALS/ EQUIPMENT

Grey cast iron as scrap, magnesium ferrosilicon was used for treating the melt, graphite (in powder form) as re-carburizer, calcium carbide ( $\text{CaC}_2$ ) used as desulphuriser and calcium trioxo carbonate (iv) salt ( $\text{CaCO}_3$ ) used as flux. Rotary furnace (100 kg capacity) for melting the scrap; ladle lined with refractory material was built with pocket for sandwich process of adding magnesium (Mg). Diesel fuel was used for firing, and inserted thermocouple with temperature controller to monitor the temperature. Optical light emission spectrometer was used for analyzing the chemical composition. Rotating disc grinder and polisher were used to prepare the metallographic samples. Universal tension meter for tensile test and Rockwell hardness tester was used to measure the hardness value of the samples.

## 3. METHODS

The composition of the scrap used was analyzed using optical light emission spectrometer and is given in Table 1. The furnace was first pre heated for 40 minutes before the scrap was charged into the furnace. Theoretically the charge was prepared with carbon percent of 3.9 and silicon of between 2.1 and 2.8 percent after inoculation. 2 kg of  $\text{CaCO}_3$  (flux) was charged to aid easy removal of slag. The furnace and its charge was heated to a temperature of 1430 °C, then 0.30 kg calcium carbide was added to the melt to reduce some sulphur from the melt in the furnace. (Calcium carbide is highly aggressive on the refractory lining of furnace, so little quantity was used). Three tapping were made one after the other and tagged A, B and C. The ladle used was made to accommodate 15 Kg liquid metal. 3.5 % of the ladle capacity (15 kg) was the weight of magnesium ferrosilicon added to A. The magnesium ferrosilicon contained 5 % Mg and 42 % silicon. 3.0 % of the ladle capacity was the Mg added to B and 2.0 % of the ladle capacity was added to C. They were cast into sand mould prepared with diameter 20.0 mm patterns. They were allowed to solidify to room temperature in the mould. The as-casts chemical compositions were determined with optical light emission spectrometer. Micro structural examination was examined using optical microscopy. It revealed that castings from A and B contain nodules that showed they are nodular iron. Tensile evaluation was done for A and B samples.

Table 1. Composition of the scrap used

C	Si	Mn	P	S	Mg
3.80	1.8	0.87	0.088	0.131	0.0033

Table 2: Chemical composition of A, nodular iron produced

C	Si	Mn	P	S	Mg
2.88	2.75	0.096	0.081	0.075	0.108

Table 3: Chemical composition of B, nodular iron produced

C	Si	Mn	P	S	Mg
2.69	2.37	0.039	0.069	0.070	0.087

from B which fits universal tester were made from the as-cast nodular iron produced. The samples were fixed one after the other into the machine and switched on. Universal Tester is an instron computerized machine which reads all the data, as tensile strain takes place according to Kenawy et al., (2001). The analyses are shown in Table 4, while the stress –strain curves for samples A and B is shown in Fig. 1.

Digital Rockwell hardness tester (C, scale with weight 150 kg) was used according to Oyetunji, (2007) and the operational guide of the machine was followed. The surfaces of the samples were prepared by grinding on emery paper of 400 grits before taking the hardness. This is to ensure flat surface on the samples so as to obtain a reliable result. According to the operation of the machine diamond indenter was pressed on the prepared surface and the results were displayed on the machine indicator in which average result was taken after taking reading from three different points on a sample.

### 3.2. Metallographic preparation

Metallographic samples of the length which can easily be held without mounting were cut out from each of the as cast-metal (A, B and C). Rotating disc grinder was used with different grit of grinding paper starting from 60, 120 400, and 600 grits. The grinded surfaces were made flat and smooth. After successful grinding operation, polishing commenced with polishing paper starting from 800grits, 1200grits and finally with polishing cloth of 3 micron with diamond paste suspension. They were polished to obtain mirror finish surfaces. After polishing they were etched with 2% nital (2% nitric acid and 98% ethanol). The etchant was applied with the aid of cotton wool for 15 seconds to attack the surface in order to reveal the structure when viewed under the microscope before taking the micrograph. Zeiss metallurgical microscope was used to take the micrograph.

4. RESULTS and DISCUSSION

Several past works and literatures have concluded that residual Mg in ductile cast iron has to be in the range of 0.03 to 0.05 % when the iron must have low sulphur of not more than 0.01 to 0.02 % . Notable among the works is Tiedje (2010) who also discussed the residual Mg to be in the range 0.03 to 0.05 % when high purity iron, carbon and silicon are used to produce the base metal. However, due to the raw material available (scrap) and the processing method (rotary furnace utilizing diesel fuel) pick up of sulphur was highly encouraged. This accounted for the ductile iron produced to contain up to 0.07% S, but we could produce nodules in the casting since we succeeded in having a residual Mg content of 0.108 % Mg in sample A and 0.087 % Mg in sample

The compositions of nodular iron produced (designated A and B) are shown in Tables 2 and 3. Tensile samples of 30 mm gauge length which fits into universal tension machine were machined on lathe machine for each sample A and B. Rockwell hardness tester was used for measuring the hardness of the as-cast samples.

### 3.1. Tensile and hardness tests

One tensile sample from casting A and one

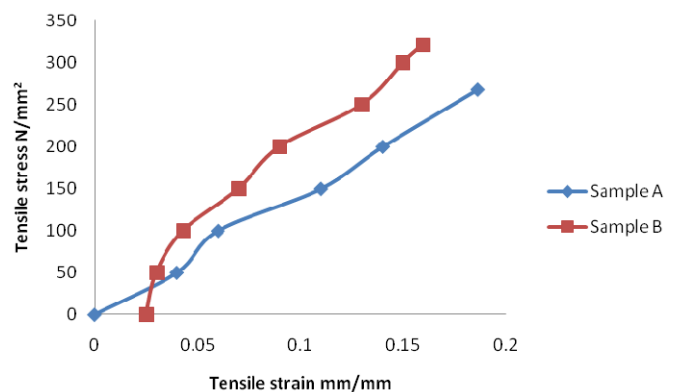


Figure 1: Stress –Strain curve for Samples A and B  
Stress –strain curve for sample A, Stress –strain curve for sample B

B. Due to the oxidation tendency of carbon in the furnace, the carbon percent has been oxidized to 2.88 and 2.69% in the casting. The amount of carbon loss through oxidation is a function of processing time spent during melting. This has reduced the % CEV of the ductile cast iron produced to 3.82 and 3.50 respectively. Thus, hypoeutectic iron was produced because it has % CEV less than 4.3% (Brown, 1994). As a result of this low CEV, the freezing range of the melt has to be shorter thereby allowing nodules to be retained in the casting without the Mg undergoing complete desulphurization of the melt before the nodules can be sustained. In line with Hassan Jafari, et al 2010 there will be heterogeneous nucleation which will lead to shorter solidification time thereby increasing the graphite nodules count. If there had been enough time for reactions and solidification, the Mg may have undergone desulphurization of the melt completely before the onset of nodularisation reaction.

Table 4: Tensile analysis of sample A and B

A Extension at max. load (mm) 5.42	A Tensile strain at max. load % 18.502	A Tensile stress at max. load MPa 268.306	B Extension at max. load (mm) 4.83	B Tensile strain at max. load % 16.54	B Tensile stress at max. load MPa 321.861
A Tensile extension at break (standard) mm 5.42	A Tensile strain at break (standard) % 18.502	A Tensile stress at break (standard) MPa 268.306	B Tensile extension at break (standard) mm 4.83	B Tensile strain at break (standard) % 16.54	B Tensile stress at break (standard) MPa 321.861
A Diameter of sample (mm) 5.82	A Area (Cm <sup>2</sup> ) 0.267	A Length of sample (mm) 28.95	B Diameter of sample (mm) 5.82	B Area(Cm <sup>2</sup> ) 0.267	B Length of sample (mm) 28.95

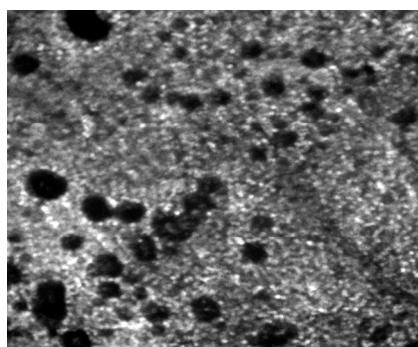
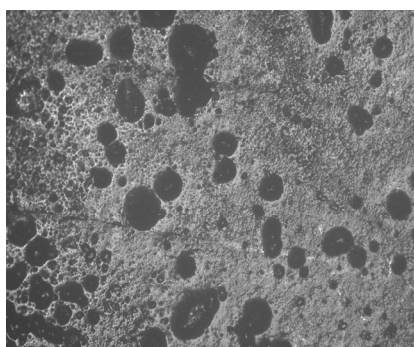
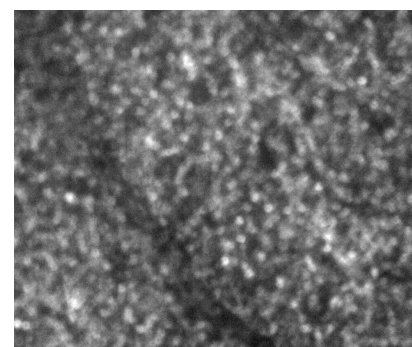
Microstructure of sample A  
(etched in 2% nital) X200Microstructure of Sample B  
(etched in 2% nital) X200Microstructure of sample C  
(etched in 2% nital) X200

Figure 2: Microstructure of samples A, B and C

There could not be significant necking before fracture despite the fact that elongation of up to 18 % was achieved. This is because the scrap used by analysis contained 0.088% phosphorus and the ductile iron produced contained 0.081 % and 0.069 % phosphorus respectively, and maximum phosphorus desirable in ductile iron should be less than 0.05 % P (Brown, 1994 and Ajuwa, 1998). This is because phosphorus is an embrittling agent which forms phosphide eutectic at the grain boundary location thereby reducing the impact property. As a result of the yet unavoidable phosphorus in the casting, at ultimate load it fractures without appreciable necking as shown in Fig. 1.

## 5. CONCLUSION

- ✓ The result has demonstrated that at higher residual magnesium content above 0.05 % and even with higher sulphur content (above 0.02 % S) nodules can still be sustained in the casting if the freezing range can be controlled.
- ✓ The freezing range can be tied to the composition of the melt particularly the % carbon and % silicon which greatly determine the carbon equivalent value.
- ✓ As a result of high % phosphorus in the as cast, at the ultimate tensile stress fracture occurred without appreciable necking as a result of brittle nature caused by phosphorus content.



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